# Transverse Compressive Stress Effect in Y-Ba-Cu-O Coatings on Biaxially Textured Ni and Ni-W Substrates

N. Cheggour, J. W. Ekin, C. C. Clickner, D. T. Verebelyi, C. L. H. Thieme, R. Feenstra, A. Goyal, and M. Paranthaman

Abstract—Electromechanical properties of yttrium-bariumcopper-oxide (YBCO) coatings on both pure Ni and Ni-5at.%W alloy rolling-assisted, biaxially-textured substrates (RABiTS) were investigated. The effect of transverse compressive stress on transport critical-current densities  $(J_c)$  was measured on samples at 76 K and self magnetic field. Transverse compressive stress can significantly degrade  $J_c$  in YBCO deposited on pure Ni RABiTS unless sufficient frictional support is provided to the sample or the substrate is given a work-hardening treatment. On the other hand, results obtained for YBCO on Ni-5at.%W alloy RABiTS show that the tolerance to transverse stress of these conductors is significantly improved. These electromechanical properties are interpreted with scanning-electron micrographs of the microstructure of the samples after electromechanical testing, as well as stress-strain characteristics measured on RABiTS substrates at 76 K. The tensile yield strength, Young's modulus, and proportional limit of elasticity of candidate RABiTS substrate materials are tabulated and compared.

Index Terms—Coated conductors, critical current density, mechanical properties, microstructure, RABiTS, strain, stress, Y-Ba-Cu-O.

#### I. INTRODUCTION

films deposited on buffered, flexible, metallic substrates exhibit critical-current densities  $(J_c)$  that are in excess of 1 MA/cm<sup>2</sup> at liquid-nitrogen temperature and self magnetic field [1]–[3]. These coated conductors may therefore potentially be used in the construction of electrical devices such as underground power-transmission lines, transformers, motors, generators, and magnetic separators. Strong grain alignment is essential for achieving such high values of  $J_c$  [4]–[9]. This requirement can be met by using, for example, rolling-assisted, biaxially-textured substrates (RABiTS) [2], [3] or ion-beam-assisted deposition (IBAD) [1]. Since the ceramic coatings are inherently brittle, the tolerance of the coated conductors to mechanical stresses can affect the ultimate performance

Manuscript received August 5, 2002. This work was supported in part by the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Office of Power Technologies—Superconductivity Program, and the U.S. Department of Energy, Division of High Energy Physics. Contribution of NIST, not subject to copyright.

- N. Cheggour, J. W. Ekin, and C. C. Clickner are with the National Institute of Standards and Technology, Boulder, CO 80305 USA (e-mail: cheggour@boulder.nist.gov; ekin@boulder.nist.gov).
- D. T. Verebelyi and C. L. H. Thieme are with American Superconductor Corporation, Westborough, MA 01581 USA.
- R. Feenstra, A. Goyal and M. Paranthaman are with the Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA.

Digital Object Identifier 10.1109/TASC.2003.812390

of devices made of these tapes. Hence, the study of their electromechanical properties is crucial to both guide conductor development and provide critical data for the design of specific applications [1], [10]–[15]. When a power-transmission cable is bent, each tape element is subjected to bending strain and also to transverse compressive stress as the tapes in the different layers of the cable are pushed against each other and against the wall of the conduit. Also in magnet applications, the Lorentz force applied to the windings radially produces a hoop stress that can transform both to axial tensile strain and transverse compressive stress in the tapes. In this work we present experimental results on the effect of transverse compressive stress on  $J_c$  obtained in YBCO films deposited on pure Ni and Ni-5at.%W *alloy* RABiTS. The microstructure of the samples after electromechanical testing reveals cracking of the ceramic films due to application of stress. We also report stress-strain characteristics of candidate RABiTS substrate materials (annealed pure Ni, Ni-13at.%Cr, Ni-3at.%W-2at.%Fe, and Ni-5at.%W) and compare their tensile yield strength, Young's modulus, and proportional limit of elasticity at 76 K. These results provide insight into the role played by the mechanical properties of the substrate in determining the electromechanical properties of these conductors, as well as provide a preliminary comparison of the effect of transverse stress on YBCO coatings on pure Ni versus Ni-5at.%W alloy substrate materials.

#### II. EXPERIMENTAL PROCEDURE

Ni and Ni-5at.%W rods were thermomechanically processed to obtain biaxially textured substrates [16], [17]. Buffer layers were grown on the substrates by reactive evaporation and RF magnetron sputtering. Thereafter, the YBCO layer was deposited on the buffered substrates using the ex situ BaF<sub>2</sub> method [18], [19]. Samples with pure Ni (Samples 1 to 3) had a structure, from outside in, of YBCO |CeO<sub>2</sub>| yttria-stabilized zirconia (YSZ) |CeO<sub>2</sub>| Ni [16]. Samples 4 and 5 incorporated a Ni-5at.%W substrate and had an architecture of YBCO [CeO<sub>2</sub>] YSZ  $|Y_2O_3|$  Ni | Ni-W [17]. The tapes were then coated with a layer of Ag and annealed in oxygen. An additional layer of Ag (total thickness around 10  $\mu$ m) was deposited by thermal evaporation to ensure low contact resistivity between the sample and the current leads after soldering connections. For Samples 1 to 3, the thickness of the pure Ni substrate was 50  $\mu$ m, that of the buffer layers less than 1  $\mu$ m, and that of the YBCO layer about 0.3  $\mu$ m. Samples 4 and 5 had a Ni-5at.%W substrate 75  $\mu$ m thick, a cap Ni layer 2  $\mu$ m thick, and buffer layers less than 1  $\mu$ m, while the thickness of YBCO layer was 1  $\mu$ m.

The coated-conductor samples had a width of about 3 mm to 4 mm and a length of about 2.5 cm. All the results reported here were obtained on samples with an initial critical-current density on the order of 1 MA/cm<sup>2</sup> at 76 K, measured with an electrical-field criterion of 1  $\mu$ V/cm in self-field.

The apparatus for measuring  $J_c$  vs. transverse stress  $(\sigma_T)$ has been described by Ekin [20]. Two copper leads and a pair of voltage taps were soldered to the sample. The sample was mounted onto a flat stainless steel block (bottom anvil) located at the bottom end of the apparatus. The current leads were designed and soldered to the apparatus in such a way that one copper lead was stationary and the other was free to flex [21]. The flexible copper lead ensured that the sample was stress-free during the cooling of the apparatus from room temperature to the operating temperature. The top anvil, also flat and made of stainless steel, was attached to the probe via a biaxially gimbaled pressure-foot so that this anvil conformed to the sample and bottom anvil surfaces. The load was transmitted to the sample via a stainless steel rod, which was attached at the top end of the probe to a calibrated 13 kN load cell to measure the force applied to the sample. Measurements were carried out in liquid nitrogen at 76 K. Voltage-current curves were taken as a function of  $\sigma_T$  up to 180 MPa in self-field, and values of  $J_c$  were determined at an electrical-field criterion of 1  $\mu$ V/cm. Uncertainties in the measurements of  $J_c$  and  $\sigma_T$  were about 1% and 2%, respectively.

The apparatus for measuring stress-strain characteristics was constructed to investigate very soft substrates [22]. The procedure for mounting samples in the apparatus was adapted to avoid work-hardening the substrates prior to measuring stress vs. strain. Substrates were 50  $\mu$ m or 75  $\mu$ m thick, 1 cm wide and 30 cm long. The apparatus was connected to a servo-hydraulic actuator equipped with a 1300 N load cell and a linear variable differential transformer (LVDT) to measure force and displacement, respectively. The apparatus was inserted, under load control, into a liquid-nitrogen cryostat to ensure stress-free cooling of the sample. To check the accuracy of the measurements, the Young's modulus was determined for stainless-steel tapes about 80  $\mu$ m thick and 3.5 mm wide and was found to be 176 GPa at 295 K, 200 GPa at 76 K, and 210 GPa at 4 K. These values are consistent with those for type 304 stainless-steel sheets. Uncertainties in determining the Young's modulus and yield strength are due mainly to the thickness, and can be estimated to about 10%, depending on the variation in thickness along the substrate's length.

### III. TRANSVERSE COMPRESSIVE STRESS RESULTS

Two modes of application of transverse compressive stress to the sample were used. In the *monotonic-loading* mode, stress was applied to the sample and gradually increased without releasing the load between measurement steps. In contrast, the *load-unload* mode consisted of applying stress of a certain value to the sample, then releasing it (with both anvils maintaining physical contact with the sample) before reapplying it at a higher value. Sample 1 and 2 were measured in the *monotonic-loading* mode and *load-unload* mode, respectively, and presented very different behavior (Fig. 1). The monotonic loading mode produced no degradation of  $J_c$  up to about

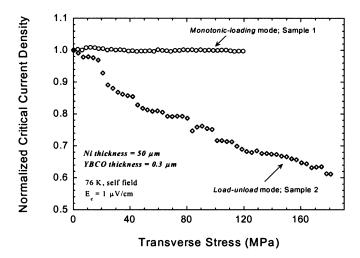


Fig. 1. Effect of transverse stress on  $J_c$  in YBCO films on *pure* Ni RABiTS. The results obtained in two modes of measurements illustrate the role played by friction between the sample and the pressing anvils.

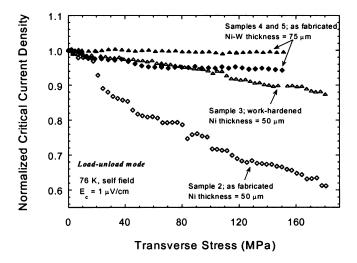


Fig. 2. Effect of transverse stress on  $J_c$  in YBCO films on *pure* Ni and Ni-5at.% W *alloy* RABiTS. The results illustrate the benefits of work-hardening pure Ni and provide a comparison between YBCO coatings on pure Ni and Ni-5at.% W alloy substrate materials.

120 MPa, whereas the load-unload mode resulted in significant degradation in  $J_c$ , amounting to about 28% at 100 MPa and 39% at 180 MPa. In the *monotonic-loading* mode, the sample has a strong frictional support from the pressing anvils. We believe this support prevents the sample from expanding laterally. In contrast, the frictional support is significantly reduced when operating in the *load-unload* mode. In-plane expansion becomes possible, which may lead to cracking of the buffer and YBCO layers.

In Fig. 2 we show the benefit of work-hardening the sample substrate and compare the response to transverse stress of the YBCO coatings on pure Ni versus Ni-5at.%W alloy substrate materials. Sample 3 was loaded first monotonically to 160 MPa, then unloaded and measured in the *load-unload* mode up to 180 MPa. In the monotonic loading,  $J_c$  did not degrade (result not shown in Fig. 2). During this operation, the pure Ni substrate work-hardened, which substantially improved the tolerance of this sample to transverse stress as compared to Sample 2, which did not receive the work-hardening treatment. Sample



Fig. 3. Scanning electron micrograph of YBCO film on buffered *pure* Ni RABiTS substrate after *transverse*-stress measurements, showing multi-patterned mechanical cracks throughout the sample, both longitudinal and transverse to the electrical-current flow direction. The vertical axis of the image coincides with the direction of the electrical current applied to the sample.

4 and 5, which had a Ni-5at.%W substrate, exhibited the best tolerance to transverse stress in the *load-unload* mode of all the samples measured (degradation < 1% for Sample 4 and < 6% for Sample 5 at 150 MPa), and this performance was obtained without a work-hardening treatment.

These results provide evidence that the mechanical properties of the substrate material play a dominant role in determining the response of these samples to transverse compressive stress. Another possible source for the degradation of  $J_c$  could be delamination of the ceramic layers due to application of stress. More comprehensive data are still required to draw definitive conclusions.

As presented below, the tensile yield strength, Young's modulus, and proportional limit of elasticity of Ni-5at.%W substrates are substantially higher than those of pure Ni substrates. The mechanical properties of work-hardened pure Ni substrates are also expected to be superior, as compared to the properties of pure Ni substrates without work hardening. For the samples measured, the higher values these properties have, the greater the transverse load required to initiate cracking of the ceramic layers. These results do not rule out the usefulness of soft substrate materials. If strong frictional support can be provided to the tape, even YBCO films on soft substrates may suffer little or no damage from transverse stress up to at least 160 MPa. Nonetheless, it is probably not convenient to provide sufficient frictional support to the tape in many applications. Therefore, it is safer to use the more robust Ni-alloy substrate materials in the coated conductors' architecture.

# IV. MICROSTRUCTURE STUDY

After electromechanical testing, silver was etched away to expose the YBCO layer. A solution of  $25\%H_2O_2+25\%NH_4OH+50\%H_2O$  was used for silver etching [14]. The microstructure of this layer was examined by scanning electron microscopy (SEM), and regions with cracks were found (Figs. 3 and 4). For samples with pure Ni substrates tested under *transverse* stress, the cracked regions were distributed in patches, with sizes from

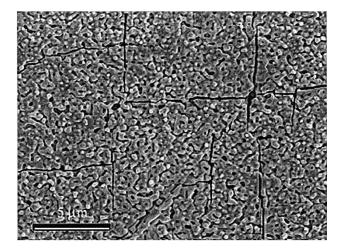


Fig. 4. Scanning electron micrograph of YBCO film on buffered Ni-5at.%W *alloy* RABiTS substrate after *transverse*-stress measurements, showing fine mechanical cracks longitudinal and transverse to the electrical-current flow direction. These cracks were rare and located mostly near the edges of the sample. The vertical axis of the image coincides with the direction of the electrical current applied to the sample.

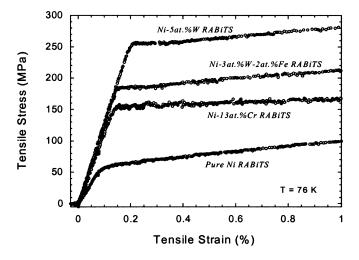


Fig. 5. Comparison of stress-strain curves at 76 K of annealed *pure* Ni, Ni-13at.%Cr, Ni-3at.%W-2at.%Fe, and Ni-5at.%W *alloy* RABiTS substrates.

a few micrometers to about 600  $\mu$ m wide. The cracks were both longitudinal as well as transverse to the direction of the electric current flow (Fig. 3). In the case of coated Ni-5at.% W substrate samples exposed to transverse pressing, cracks were rare, located mostly near the sample edges (Fig. 4). Formation of cracks in the stressed ceramic layers is the primary reason for the degradation of  $J_c$ .

## V. MECHANICAL PROPERTIES OF PURE NI AND NI ALLOY RABITS SUBSTRATES AT 76 K

Stress-strain characteristics were measured for *pure* Ni, Ni-13at.%Cr, Ni-3at.%W-2at.%Fe and Ni-5at.%W *alloy* RABiTS substrates at 76 K (Fig. 5). The samples investigated received the same heat treatment as those used for buffer and YBCO depositions. Several samples of each material were measured. Average values of the tensile yield strength, Young's modulus, and proportional limit of elasticity are summarized in Table I. The tensile yield strength of Ni-5at.%W is higher than

TABLE I
MECHANICAL PROPERTIES AT 76 K OF RABITS MATERIALS

	$\varepsilon_{p}$ (%)	σ <sub>γ</sub> (MPa)	E (GPa)
Pure Ni	0.08	59	72
Ni-13at.%Cr	0.15	157	104
Ni-3at.%W-2at.%Fe	0.15	183	128
Ni-5at.%W	0.2	254	128

 $\varepsilon_p$ : Proportional limit of elasticity.

 $\sigma_{\gamma}$ : Tensile yield strength; 0.02 % offset criterion.

E: Effective Young's modulus (initial slope of stress vs. strain).

that of pure Ni by more than a factor of 4, whereas the Young's modulus and proportional limit of elasticity are respectively higher by factors of 1.8 and 2.5. Also note that in the plastic regime, the stress increases with strain at a higher rate for pure Ni than for the measured Ni alloy RABiTS materials. Therefore the work-hardening of pure Ni is greater than that of Ni-W or Ni-Cr substrates.

## VI. CONCLUSION

The degradation of transport critical-current density from transverse compressive stress in YBCO coated conductors on buffered pure Ni and Ni-5at.% W alloy RABiTS has been investigated at 76 K and self-magnetic field. Transverse compressive stress can significantly degrade  $J_c$  in YBCO deposited on pure Ni RABiTS unless sufficient frictional support is provided to the sample. Appropriate work hardening of pure Ni substrates or the use of Ni-5at.% W alloy substrates improves the tolerance of these conductors to transverse compression.

The microstructure of the samples after electromechanical testing has shown cracking of the YBCO layer. These cracks are the primary cause for the degradation of  $J_c$  due to stress.

The mechanical properties of annealed pure Ni, Ni-13at.%Cr, Ni-3at.%W-2at.%Fe, and Ni-5at.%W alloy RABiTS substrates at 76 K have also been measured and tabulated. These results are given to guide the development of YBCO coated conductors.

# REFERENCES

- X. D. Wu, S. R. Foltyn, P. N. Arendt, W. R. Blumenthal, I. H. Campbell, J. D. Cotton, J. Y. Coulter, W. L. Hults, M. P. Maley, H. F. Safar, and J. L. Smith, "Properties of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> thick films on flexible buffered metallic substrates," *Appl. Phys. Lett.*, vol. 67, pp. 2397–2399, 1995.
- [2] A. Goyal, D. P. Norton, J. D. Budai, M. Paranthaman, E. D. Specht, D. M. Kroeger, D. K. Christen, Q. He, B. Saffian, F. A. List, D. F. Lee, P. M. Martin, C. E. Klabunde, E. Hartfield, and V. K. Sikka, "High critical current density superconducting tapes by epitaxial deposition of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> thick films on biaxially textured metals," *Appl. Phys. Lett.*, vol. 69, pp. 1795–1797, 1996.
- [3] D. P. Norton, A. Goyal, J. D. Budai, D. K. Christen, D. M. Kroeger, E. D. Specht, Q. He, B. Saffian, M. Paranthaman, C. E. Klabunde, D. F. Lee, B. C. Sales, and F. A. List, "Epitaxial YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> on biaxially textured nickel (001): An approach to superconducting tapes with high critical current density," *Science*, vol. 274, pp. 755–757, 1996.

- [4] D. Dimos, P. Chaudhari, J. Mannhart, and F. K. LeGoues, "Orientation dependence of grain-boundary critical currents in YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> bicrystals," *Phys. Rev. Lett.*, vol. 61, pp. 219–222, 1988.
- [5] D. Dimos, P. Chaudhari, and J. Mannhart, "Superconducting transport properties of grain boundaries in YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> bicrystals," *Phys. Rev. B*, vol. 41, pp. 4038–4049, 1990.
- [6] Y. Iijima, N. Tanabe, O. Kohno, and Y. Ikeno, "In-plane aligned YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> thin films deposited on polycrystalline metallic substrates," *Appl. Phys. Lett.*, vol. 60, pp. 769–771, 1992.
- [7] A. Goyal, D. P. Norton, D. M. Kroeger, D. K. Christen, M. Paranthaman, E. D. Specht, J. D. Budai, Q. He, B. Saffian, F. A. List, D. F. Lee, E. Hatfield, P. M. Martin, C. E. Klabunde, J. Mathis, and C. Park, "Conductors with controlled grain boundaries: An approach to the next generation, high temperature wire," J. Mater. Res., vol. 12, pp. 2924–2940, 1997.
- [8] D. T. Verebelyi, D. K. Christen, R. Feenstra, C. Cantoni, A. Goyal, D. F. Lee, M. Paranthaman, P. N. Arendt, R. F. DePaula, J. R. Groves, and C. Prouteau, "Low angle grain boundary transport in YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> coated conductors," *Appl. Phys. Lett.*, vol. 76, pp. 1755–1757, 2000.
- [9] D. T. Verebelyi, C. Cantoni, J. D. Budai, D. K. Christen, H. J. Kim, and J. R. Thompson, "Critical current density of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> low-angle grain boundaries in self-field," *Appl. Phys. Lett.*, vol. 78, pp. 2031–2033, 2001
- [10] H. C. Freyhardt, J. Hoffmann, J. Wiesmann, J. Dzick, K. Heinemann, A. Isaev, F. Garcia-Moreno, S. Sievers, and A. Usoskin, "YBaCuO thick films on planar and curved technical substrates," *IEEE Trans. Appl. Supercond.*, vol. 7, pp. 1426–1431, 1997.
- [11] C. Park, D. P. Norton, J. D. Budai, D. K. Christen, D. T. Verebelyi, R. Feenstra, D. F. Lee, A. Goyal, D. M. Kroeger, and M. Paranthaman, "Bend strain tolerance of critical currents for YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> films deposited on rolled-textured (001) Ni," *Appl. Phys. Lett.*, vol. 13, pp. 1904–1906, 1998.
- [12] C. L. H. Thieme, S. Fleshler, D. M. Buczek, M. Jowett, L. G. Fritze-meier, P. N. Arendt, S. R. Foltyn, J. Y. Coulter, and J. O. Willis, "Axial strain dependence at 77 K of the critical current of thick YBaCuO films on Ni-alloy substrates with IBAD buffer layers," *IEEE Trans. Appl. Supercond.*, vol. 9, pp. 1494–1497, 1999.
- [13] J. Yoo and D. Youm, "Tensile stress effects on the critical current densities of coated conductors," *Supercond. Sci. Technol.*, vol. 14, pp. 109–112, 2001.
- [14] J. W. Ekin, S. L. Bray, N. Cheggour, C. C. Clickner, S. R. Foltyn, P. N. Arendt, A. A. Polyanskii, D. C. Larbalestier, and C. N. McCowan, "Transverse stress and fatigue effects in Y-Ba-Cu-O coated IBAD tapes," *IEEE Trans. Appl. Supercond.*, vol. 11, pp. 3389–3392, 2001.
- [15] N. Cheggour, J. W. Ekin, C. C. Clickner, R. Feenstra, A. Goyal, M. Paranthaman, D. F. Lee, D. M. Kroeger, and D. K. Christen, "Transverse compressive stress, fatigue, and magnetic substrate effects on the critical current density of Y-Ba-Cu-O coated RABiTS tapes," Adv. Cryo. Eng., vol. 48, pp. 461–468, 2002.
- [16] A. Goyal, R. Feenstra, M. Paranthaman, J. R. Thompson, B. Y. Kang, C. Cantoni, D. F. Lee, F. A. List, P. M. Martin, E. Lara-Curzio, C. Stevens, D. M. Kroeger, M. Kowalewski, E. D. Specht, T. Aytug, S. Sathyamurthy, R. K. Williams, and R. E. Ericson, "Strengthened, biaxially textured Ni substrate with small alloying additions for caoted conductor applications," *Physica C*, vol. 382, pp. 251–262, 2002.
- [17] D. T. Verebelyi, U. schoop, C. L. H. Thieme, S. Annavarapu, S. Cui, X. Li, W. Zhang, T. Kodenkandath, L. Fritzemeier, Q. Li, A. P. Malozemoff, N. Nguyen, D. Buczek, J. Lynch, J. Scudiere, and M. Rupich, Uniform Performance of Meter Long YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-6</sub> Superconductor on Textured Ni Alloy Substrates: American Superconductor Corporation.
- [18] R. Feenstra, T. B. Lindemer, J. D. Budai, and M. D. Galloway, "Effect of oxygen pressure on the synthesis of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> thin films by postdeposition annealing," *J. Appl. Phys.*, vol. 69, pp. 6569–6585, 1991.
- [19] P. C. McIntyre, M. J. Cima, and A. Roshko, "Epitaxial nucleation and growth of chemically derived  $Ba_2YCu_3O_{7-x}$  thin films on (001)  $SrTiO_3$ ," *J. Appl. Phys.*, vol. 77, pp. 5263–5272, 1995.
- [20] J. W. Ekin, "Effect of transverse compressive stress on the critical current and upper critical field of Nb<sub>3</sub>Sn," *J. Appl. Phys.*, vol. 62, pp. 4829–4834, 1987.
- [21] P. E. Kirkpatrick, J. W. Ekin, and S. L. Bray, "A flexible high-current lead for use in high-magnetic-field cryogenic environments," *Rev. Sci. Instrum.*, vol. 70, pp. 3338–3340, 1999.
- [22] C. C. Clickner, J. W. Ekin, and N. Cheggour, National Institute of Standards and Technology, Boulder, CO, unpublished.